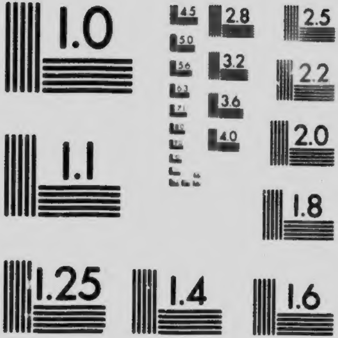

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UNIVERSITY OF TORONTO STUDIES

PAPERS FROM THE CHEMICAL LABORATORIES

No. 68: THE ESTIMATION OF BORIC ACID AND BORATES IN FOODSTUFFS AND COMMERCIAL PRODUCTS,

BY R. J. MANNING AND W. R. LANG.

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BY
R. J. MANNING AND W. R. LANG.

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THE ESTIMATION OF BORIC ACID AND BORATES IN FOODSTUFFS AND COMMERCIAL PRODUCTS.

BY R. J. MANNING AND W. R. LANG.

In a previous paper (Manning and Lang; this J., 1906, 397), a new method of estimating boric acid was described, which was found to give results in some cases to within 0·05% of the theoretical. Since the completion of this work the method has been used in the University Laboratory of Applied Chemistry and pronounced satisfactory Its application to the determination of borates in such articles as milk, common salt, etc., is the object of this present paper, these substances being found to contain small quantities of boric acid in some form or other.

The results of this investigation are now given arranged as follows:—

(1) Two methods for the determination of borates in milk.

(2) Two methods for their determination in common methylated spirits.

(3) Two methods for their determination in commercial common salt.

ESTIMATION OF BORIC ACID IN MILK.

Method I.—Distillation and precipitation as barium borate.—This method is almost exactly similar to that previously published (*loc. cit.*), only, of course, adapted to the needs of the case. The first precaution necessary was to obtain milk absolutely free from boric acid and borates. About 300 c.c. of pure milk was introduced into a distilling flask and some boric acid dissolved in it, or else it was introduced in the alcohol, of which 400 c.c. were at first taken. Additions of more alcohol were made from time to time to ensure the complete distillation of the tri-ethyl or tri-methyl borate. It was now found that no matter how much boric acid was introduced in the milk that no precipitate whatever of barium borate was formed. In order to have the tri-methyl borate formed the introduction of sulphuric acid was necessary. A long series of experiments were therefore undertaken to determine what amount of sulphuric acid was necessary with 300 c.c. of milk, as a result of which it was found that at least 70 c.c. of concentrated sulphuric acid are necessary; the large amount of water in the milk decomposes the tri-methyl compound into boric acid and alcohol. In this case the boric acid will distil over only at a very high temperature. The sulphuric acid appears to prevent this decomposition. After distilling for half an hour, the distillate was treated with a few drops of a concentrated solution of barium chloride. At this stage a slight precipitate sometimes occurred. The fats in the milk contain three esters, those of palmitic, stearic, and oleic acids. These all form compounds with barium chloride which are insoluble in water and in cold dilute hydrochloric acid. It was found that if the temperature in the distilling flask rose about 80° C., a small portion of these (probably oleic acid) was carried over. Therefore, on the addition of barium chloride, a flocculent precipitate of barium oleate appeared. This had to be removed by filtration before the experiment could be proceeded with. The distillate was now titrated against a freshly prepared concentrated sodium hydroxide solution, prepared, as before described, by dissolving newly cut pieces of sodium in water in a silver dish; phenolphthalein was used as indicator. Care had to be taken to guard against overstepping the neutral point, as the excess of the alkali united with the barium chloride to form the hydroxide. The precipitate of barium borate was now filtered off on a tared filter paper, washed carefully with alcohol, dried at 110° C., and weighed.

First of all a test series was made in order to see how small a quantity of boric acid would, on neutralisation of the distillate, give a precipitate distinctly seen by the naked eye. To this end, 50, 40, 30, 20, 10 parts boric acid were introduced into 10,000 parts of milk. The precipitate of 50 parts boric acid in 10,000 parts milk was quite heavy, the 40 less so. In this manner a gradation was obtained until when 10 parts in 10,000 was reached only a faint milkiness was obtained. This last was repeated several times with precisely the same result. Below this point traces of precipitate became untrustworthy, especially as a trace of boron compounds could be obtained from impure methylated spirits. This, however, will be dealt with later.

The results obtained from weighed precipitates were :—

Boric acid introduced.	Barium borate.	Yield.
grms.	grms.	Per cent.
1·50	2·6890	99·5
1·50	2·6830	99·3
0·50	0·8941	99·25
0·15	0·2649	98·0
0·12	0·1970	91·1
0·09	0·1414	87·2
0·06	0·0879	81·3
0·03	0·0340	63·33

Method II.—Distillation and titration.—Instead of precipitating the boric acid from the distillate as in Method I., the distillate was diluted with water up to one litre. Portions of 100 c.c. were neutralized with $N/10$ potassium hydroxide in presence of phenolphthalein. About 30 c.c. of glycerin were then added, and the titration continued until a permanent shade of pink was obtained.

Boric acid introduced.	Yield.
grms.	Per cent.
1·50	99·6
1·50	99·5
0·50	99·47
0·15	98·43
0·12	92·1
0·09	88·7
0·06	83·0
0·03	69·2

Method III.—The milk was first evaporated and afterwards charred. The charred mass was then treated with water and titrated in the presence of glycerin against potash, using phenolphthalein as indicator. The method is not yet complete and the results are temporarily withheld.

ESTIMATION OF BORATES IN METHYLATED SPIRITS.

Method I.—By distillation and precipitation.—The method was exactly similar to that used in the case of milk; 300 c.c. of methylated spirits and 25 c.c. of concentrated sulphuric acid were distilled until the temperature rose to 75°, when the boron compound was precipitated by barium chloride and weighed as before. One litre of methylated spirits was found to contain 0·0183 grm. boric acid as a borate of some sort.

Method II.—By distillation and titration.—Owing to the exceedingly small amounts of boric acid in the alcohol it was very difficult to obtain results that agreed. The large distillate was diluted to a known volume (500 c.c. usually), and portions of 100 c.c. taken out at a time and titrated against $N/10$ potassium hydroxide with 30 c.c. glycerin and a few drops of phenolphthalein. One litre of methylated spirits was found to contain 0·0160 grm. boric acid.

ESTIMATION OF BORATES IN SALT.

Method I.—By Distillation and Precipitation.—A weighed amount of common packing salt (preferably about 5 grms.) were dissolved in the necessary amount of concentrated sulphuric acid. This salt solution was now introduced with 300 c.c. of methylated spirit into a distilling flask. From this point on, the experiments were exactly similar to the previous ones. From the estimations it was found that 100 grms. salt contain 0·88 grm. of boric acid as a borate.

Method II.—By Distillation and Titration.—Instead of precipitating the boric acid as barium borate, it was titrated against $N/10$ potassium hydroxide in the usual manner. 100 grms. of packing salt were found to contain 0·874 grm. of boric acid as a borate.

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